Appln. S.N. 10/673,762
Exhibit 1 for Amdt. dated August 9, 2006
Reply to Office Action of May 9, 2006
Docket No. GP-302388-R&D-KM

Exhibit 1:

Table 1
Properties of cubic boron nitride, diamond and diamond-like carbon

Property nomenclature	Cubic boron nitride c-BN	Diamond	Diamond-like carbon DLC, i-C(Me), a-C:H, Me-C:H
Chemical composition	50 at% B, 50 at% N	>99% C (impurities)	20–50 (at% H) 0–35 at% Me (Fe, W, Ta, Ti, Si, Cr, ...) Balance: C
Lattice structure	Cubic, $a=0.3616$ nm	Cubic, $a=0.3567$ nm	Amorphous (microcrystalline)
Type of bonding	sp^3	sp^3	sp^2, sp^3, sp
Hardness	5500–7000 HV	7000–10 000 HV	1500–6000 HV
Young's modulus	700 GPa	1000 GPa	
Thermal conductivity	2 W cm^{-1} K^{-1}	20 W cm^{-1} K^{-1} (Cu: 4 W cm^{-1} K^{-1})	
Phase transformation	hex., restricted	hex. (graphite)	
Temperature stability	Oxidation stability: $T \leq 1400$ °C Protective B_2O_3 layer	Oxidation stability: $T < 800$ °C Volatile CO_2 Graphitization	$T < 250$ °C Graphitization

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Tribological properties of diamond-like nanocomposite coatings at high temperatures

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Abstract

Diamond-like nanocomposites (DLN) films are amorphous and consist of C, H, Si, and O. In addition, metal atoms can be incorporated into the coatings. The DLN coatings exhibit a low coefficient of friction, low wear rate, low stress, high hardness and good adhesion on a variety of substrates. The thermal stability of DLN films deposited on silicon substrates was investigated after the samples had been thermally annealed in air and argon atmospheres. The sample analysis by micro-Raman spectroscopy, nano-indentation and ball-on-disk tribometry are reported here. The investigation revealed that the performance of DLN films from a tribological perspective is very good up to 400°C in air. The hardness of the DLN films showed a decrease of less than 15% up to 400°C in air. The wear rate of the DLN films was at or below 10^{-7} mm^3/Nm up to 400°C in air.

Author Keywords: Diamond-like nanocomposites; DLN; High temperatures; Doped diamond-like carbon; Tribological properties

Article Outline

1. Introduction

Diamond-like carbon films are comprised of amorphous carbon-hydrogen bonds (a-C:H), which exhibit sp^3 hybridization. DLC is of interest as an inexpensive alternative to diamond for many industrial applications due to its diamond-like properties. These properties include high hardness [1], low coefficient of friction [2], high wear resistance [2], high chemical stability, high electrical resistivity [3 and 4] and high thermal conductivity [5]. However, DLC films have several known limitations, such as high internal stress [6] and low thermal stability [7].

Many new variants of diamond-like carbon films have evolved recently. Amorphous carbon films are being deposited with silicon [8, 9, 10 and 11], oxygen [10 and 11], fluorine [12], boron [13], nitrogen [13 and 14] and metals [6, 15 and 16]. Diamond-like Nanocomposites [DLN or Dylyn(r)] belong to this new family of diamond-like films and are produced by Advanced Refractory Technologies (ART).

DLN films possess an amorphous structure and consist of diamond-like (a-C:H) and quartz-like (a-Si:O) networks [11]. These atomic networks give DLN its unique mechanical, optical, chemical, thermal and electrical properties. The properties of DLN can be tailored over wide ranges to meet specific application requirements. One method of achieving tailorability is by introducing metal atoms into the amorphous DLN matrix.

DLN films possess tribological properties, such as very low friction, high endurance, high hardness, low stress, and excellent adhesion to a variety of substrates, which are important for applications aimed at reducing wear. DLN has proven to be highly wear-resistant, having an average wear rate that ranges from 1.0×10^{-7} to 1.0×10^{-8} mm^3/Nm. Another advantageous characteristic of DLN is the high thermal stability of the films, which makes them suitable candidates for many "high temperature" applications.

DLC films, which have been investigated for their thermal stability by Raman spectroscopy, FTIR, Auger electron spectroscopy and x-ray photoelectron spectroscopy, have a reported stability of only 250–300°C [7]. Ageev et al. [17] demonstrated that the stability of the film is affected by the metal dopant. They found that a high melting temperature transition metal dopant, such as tungsten, enhanced the film stability, whereas a noble metal dopant decreased

the film stability. Benlahsen et al. [18] have investigated the evolution of hydrogen as a function of annealing and its influence on the film stress. Muller et al. [10 and 12] have studied the stability of fluorinated and silicon doped hydrogenated carbon films by Raman spectroscopy. Gangopadhyay et al. [19] have studied the thermal stability of silicon doped amorphous hydrogenated carbon films from the perspective of hardness, elastic modulus, friction and wear measurements. Except for the work of Gangopadhyay et al., other research efforts have focused on analyzing the effects of annealing on crystal structure and composition. Since □diamond-like coatings□ are being considered for tribological applications, a comprehensive investigation to study the effect of annealing on the mechanical properties, such as hardness, elastic modulus and wear resistance, would be useful. The work reported by Gangopadhyay et al. does address the effect of annealing on the mechanical properties but only to a limited extent.

In this paper, we report a detailed study of the thermal stability of DLN films deposited on silicon substrates and annealed at various temperatures by studying their structural and mechanical properties. A measure of the DLN thermal stability is defined, based on mechanical properties (hardness and wear rate), and this measure is used to evaluate the thermal stability of DLN films. The stability of the mechanical properties as a function of temperatures is used as a basis to evaluate the thermal stability of DLN films. Such an investigation would be useful to determine the maximum temperature that DLN coatings (for that matter other diamond-like films also) can withstand and be effective in □high-temperature□ tribological applications.

One potential application of DLN, which would require high thermal stability, low coefficient of friction and low wear, is automotive engine components such as pistons, intake valves and valve seat inserts.

2. Experimental

2.1. Film deposition

The DLN coatings were synthesized by a plasma-enhanced deposition process [4]. A proprietary siloxane precursor was used for film deposition. Substrates were biased from 100 to 500 V, using pulsed d.c. or high-frequency fields (100–450 kHz). Magnetron sputtering was utilized to introduce metal dopants in DLN coatings. Titanium and tungsten were the dopant materials investigated.

Two methods were employed to clean the substrates prior to deposition. First, the substrates were ultrasonically cleaned with conventional chemical solvents. This was followed by in-situ plasma etching with argon gas. During DLN deposition, the substrate temperature did not exceed 150–200°C. The growth rate of the DLN films ranged from 0.5 to 1.5 [×] small mu, Greek m/h.

2.2. Thermal stability tests

For these experiments, the DLN samples used to investigate the various annealing temperatures all came from the same deposition run. The coated substrates were cut into square (2.5 cm×2.5 cm) pieces for annealing. An annealing temperature range of 200–600°C was investigated in air. The samples were annealed (held at temperature) in a Lindberg box

furnace for 2 h at each temperature. A bare silicon substrate was included in each annealing run to monitor the changes to the silicon substrate.

A second batch of samples, cut into 1 cm×2.5 cm pieces, were annealed in a Lindberg box furnace in an air atmosphere at a constant temperature of 500°C for different lengths of time. The annealing duration ranged from 2 to 10 h at 500°C.

The thermal stability of DLN in an argon atmosphere was also investigated. The DLN-coated silicon samples were annealed in a vacuum furnace with flowing argon gas for 2 h. The temperature range investigated was from 200 to 1200°C.

2.3. Characterization

The thermal stability of the DLN coating was studied using several characterization techniques. A tribometer was used to perform wear tests, a nano-indentor was used for hardness, a Raman microprobe was used to study the structure of the DLN film, and a profilometer was used for thickness measurement and wear-rate determination.

The wear rates of the annealed DLN films were measured in air at room temperature with a relative humidity of ~35% using a ball-on-disk Spire SPI-500-D Tribotester. For each test, a 5 or 10 N load was used on a clean 0.64-cm-diameter tungsten carbide ball rotating at a velocity of 225 rpm over a non-lubricated surface. The duration of each test was 10 000 revolutions. The wear rate was calculated using the cross-sectional wear track area, the length of one revolution, the total distance traveled and the load on the ball. The cross-sectional area of a wear track was obtained by stylus profilometry. To verify the experimental results, each sample was tested at two different wear track radii (0.79 and 0.95 cm). The corresponding sliding speeds were 0.19 and 0.22 m/s.

The film thickness and cross-sectional area of the wear tracks were measured using a Tencor P-10 surface profiler. To obtain thickness data representative of the whole film, the average of three different measurements was used to determine the film thickness. To calculate the cross-sectional wear track area, an average of four readings was used.

The mechanical properties of the coatings were measured by a load and displacement indentation test performed on a Nano Indentor IIs (by Nano Instruments). Hardness data (GPa) were obtained as a function of depth (nm). For each sample, five indents were measured and then averaged. From the averaged hardness data, the hardness value at 150 nm was taken to be the film hardness. The tests were performed on silicon-coated substrates, and the film thickness had to be at least 0.8 [small mu, Greek]m in order to provide an accurate hardness reading (substrate effects are observed in films less than 0.8 [small mu, Greek]m thick on silicon substrates). Bare silicon samples were included as controls in each set of indents.

A 514-nm argon ion laser excitation source and a Renishaw System 1000 micro-Raman spectrometer, having a power density of 3.4 kW/cm^2, were utilized to obtain Raman measurements. The Raman spectra were curve-fitted using the GRAMS/386 (Galactic Industries Corporation) software package. The Raman spectra were fitted with two gaussian peaks identified as the D and G peaks. Prior to curve fitting, a linear or cubic background

was subtracted from the spectra.

3. Results

3.1. Raman spectroscopy

The Raman spectra of samples obtained before and after annealing, with the background subtracted, in air at atmospheric pressure and in an argon environment are shown in Fig. 1 and Fig. 2, respectively. The individual plots have been offset for clarity. The Raman spectra show a characteristic broad amorphous peak in the as-deposited condition. The single broad peak can be deconvoluted into D and G Gaussian peaks. The integral area (ID and IG) under the D and G peaks is determined by curve-fitting. In both environments, there was an increase in the ID/IG ratio as the annealing temperature increased. Annealing at still higher temperatures resulted in separation/resolution of the D and G peaks, indicating a decrease in disorder and onset of graphitization in the DLN films. During this process, phase transformations from a disordered diamond-like structure to an ordered graphitic structure takes place. Graphitization leads to loss in film hardness and wear resistance. The samples annealed in air at atmospheric pressure showed the onset of graphitization (significant increase in the ID/IG ratio) around 400°C and a clear separation/resolution of the D and G peaks by 450°C. The separation/resolution of the D and G peaks and significant increase in the ID/IG ratio were not noticeable until 600°C for the samples annealed in argon. The Raman spectra for untreated bare silicon and annealed bare silicon are almost identical, indicating that the silicon substrates are unaffected by annealing in either air or argon environments.

(14K)

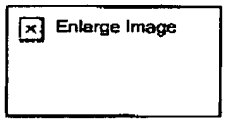
Fig. 1. Raman spectra of DLN film as a function of annealing temperature in an air atmosphere.

(13K)

Fig. 2. Raman spectra of DLN film as a function of annealing temperature in an argon astmosphere.

3.2. Hardness

Hardness data were obtained for all samples in air. For comparison, bare silicon and as-deposited samples were also included. The relationship between the hardness of the coating and the annealing temperature is shown in Fig. 3. The as-deposited DLN films had hardness values at a depth of 150 nm ranging from 12 to 18 GPa. The plot shows that the hardness remained relatively constant for most of the DLN films up to 400°C and that the hardness of the bare silicon standard was not affected by the annealing. Above 400°C, there was a sharp drop off in the film hardness.



(13K)

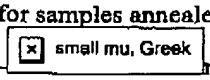
Fig. 3. Hardness of DLN films, at an indentation depth of 150 nm, as a function of annealing temperature, in air for 2 h.

3.3. Wear rate

The wear rates of the as-deposited DLN samples ranged from 1.0×10^{-8} to 1.0×10^{-7} mm^3/Nm. The results of the wear tests before and after annealing in air at atmospheric pressure are shown in Fig. 4 for undoped DLN films. In general, wear rates below 1.0×10^{-6} mm^3/Nm are considered good for a tribological coating. From this plot, it is clear that the wear rates remain relatively constant up to 400°C for all samples tested and mostly below 1.0×10^{-7} mm^3/Nm. Similar results were obtained with Ti-doped DLN.

(5K)

Fig. 4. Wear rate of DLN films as a function of annealing temperature in air.

The cross-section of the wear track (see Fig. 5), as detected by profilometry, changes significantly for samples annealed at elevated temperatures. The wear track of an as-deposited 2.3- 

m-thick DLN sample is 'V-shaped'. However, the wear track after annealing at 500°C is square-shaped. This suggests that the top layers of the 500°C annealed surface, which are graphitized/oxidized, are worn away easily, until the tungsten carbide ball reaches the underlying non-oxidized, wear-resistant DLN material.

Enlarge Image (5K)

Fig. 5. Comparison of the cross-section of the wear track for a 2.3-small mu, Greek m-thick DLN film after annealing at 200 and 500°C.

3.4. Thickness

The film thickness was measured before and after annealing the samples in air. DLN films with a thickness in the range of 0.9–5.6 small mu, Greek m were evaluated. There was a thickness measurement uncertainty of ±5%. The percentage change in thickness versus annealing temperature of undoped and metal-doped DLN films is plotted in Fig. 6.

Enlarge Image (13K)

Fig. 6. Percentage change in thickness as a function of annealing temperature in air.

Up to 350°C, the relative change in thickness of the DLN and Ti-DLN films is not significant and is within the thickness measurement uncertainty (±5%). Between 350 and 500°C, there is an onset of film attrition by graphitization/oxidation. The Ti-DLN films yield the smallest change in film thickness, less than 10% at 500°C. The DLN films, depending on their hardness, exhibit a thickness change in the range of 10–35% at 500°C.

For W-DLN films, it is observed that as the annealing temperature increases, the film attrition starts at temperatures as low as 200°C. W-DLN films show the largest change (25–55%) in film thickness at 500°C.

Also, at higher temperatures, non-uniformity in the film thickness is apparent from the observed color variations, due to interference effects. The cause of this non-uniformity in film attrition is unclear. Factors that influence this could be the uniformity of the as-deposited film, temperature gradients in the furnace, oxygen partial pressure gradients in the furnace and kinetics of the graphitization/oxidation process. This non-uniformity at □high temperatures□ causes approximately 10% uncertainty in the thickness measurement, which explains the apparent increase in thickness at higher temperatures.

A second set of samples were selected to investigate the change in thickness with different annealing times at 500°C. The thickness of the as-deposited DLN films ranged from 0.5 to

2.8 [small mu, Greek]m. The plot of the percentage change in thickness as a function of annealing duration is shown in Fig. 7. After 2 h of annealing at 500°C, there is a drastic decrease in film thickness. After 6 h at 500°C, the graphitization/oxidation process reaches a steady state, as the thickness of the film remains relatively constant. Depending on the film composition, there is a loss of film thickness from 30 to 50%. For the undoped DLN films, the residual film on the substrates has been investigated by X-ray photoelectron spectroscopy. The primary constituents of the remnant film are silicon and oxygen, with stoichiometry approaching that of silicon dioxide. This is consistent with oxidation of carbon and hydrogen during the □high-temperature□ annealing process in air.

[Enlarge Image] (6K)

Fig. 7. Plot of the percentage change in thickness as a function of annealing duration.

4. Discussion and conclusions

Raman spectroscopy of annealed DLN films indicates that the onset of structural transformation is between 200 and 400°C in air. The Raman results also indicate complete destruction of DLN coatings at 800°C in air. Further investigation in the temperature range of 200–400°C is required to narrow the transformation temperature range in air. The onset of structural transformation in a non-oxidizing atmosphere is between 400 and 600°C. In argon environments, DLN films can survive annealing temperatures up to 1100°C. As in the case of air, further investigation in the range of 400–600°C is required for an argon atmosphere to narrow the transformation temperature range. The increase in the ID/IG ratio, both in air and in an argon atmosphere, as a function of annealing temperature indicates increasing graphite dominance.

Even though Raman spectroscopy (Fig. 1 and Fig. 2) indicates that there is an onset of structural transformation between 200 and 400°C in air and 400 and 600°C in argon in DLN films, the true measure of the diamond-like performance of the coating is apparent only in the hardness and wear rate measurements.

For the purposes of this investigation, thermal stability is defined as the maximum temperature at which there is less than a 15% loss in the hardness of the DLN coating. It will become apparent that this is a consistent measure by the end of this section. Fig. 8 shows the thermal stability, as defined above, versus the as-deposited DLN film hardness. Thus, a relatively hard DLN film with an as-deposited hardness of 17 GPa, exhibits a much higher thermal stability (~400°C) than a softer, 7-GPa, DLN coating (~250°C). Fig. 3 and Fig. 8 also indicate that using the above definition, DLN films with hardness in the range of 12–18 GPa are thermally stable in the temperature range of 375–400°C.

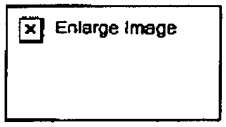


Fig. 8. Thermal stability (see text for definition) versus hardness of as-deposited DLN films after annealing in air for 2 h.

The wear tests (Fig. 4) on the annealed samples indicate that DLN films (with hardness >12 GPa) are highly wear-resistant (wear rates better than or close to 1.0×10^{-7} mm^3/Nm) even in an oxidizing environment up to 400°C. This is consistent with observations made earlier from the hardness measurements. This consistency justifies the definition of thermal stability made earlier and the corresponding assumption of 15% loss in hardness. This is further reinforced by the relatively linear relationship between wear rate and measured hardness, as shown in Fig. 9. This implies that thermal stability, as defined by the hardness criterion, is a good measure for evaluating the wear performance of □diamond-like coatings at high temperatures.□

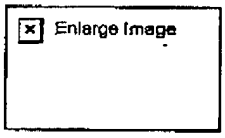


Fig. 9. Wear rate versus DLN film hardness for each annealing tempeature. The plot shows that the DLN films have wear rates between $\sim1.0\times10^{-7}$ and 1.0×10^{-8} mm^3/Nm below 400°C.

The thickness measurements as a function of annealing temperature (Fig. 6) are also consistent with the thermal stability criterion defined earlier. The thickness results indicated that in the case of both DLN and Ti-DLN films, there is minimal oxidation of the coating at least up to 350°C. An extended annealing study reveals that the film thickness decreases and reaches a steady state after 6 h at 500°C. It may be worthwhile to perform such extended annealing studies at temperatures below 500°C, to determine whether the graphitization/oxidation kinetics are slower.

Further testing is needed to determine the effects of annealing on the stability of metal-doped DLN. All W-DLN films performed poorly from a thermal stability perspective. However, this was not the case with Ti-DLN films. Some Ti-DLN films exhibited the potential to mitigate oxidation (Fig. 6 shows less than 10% loss in thickness) at □high temperatures,□ whereas others showed no significant benefit (Fig. 7).

This investigation focused on the hardness and wear rate of air-annealed DLN films as property/performance measures. A similar investigation would be useful for argon environments. It would also be worthwhile to investigate the friction coefficient of DLN coatings in □high-temperature□ environments.

Finally, Raman spectroscopy indicates that there is an onset of structural transformation in DLN and Ti-DLN films between 200 and 400°C in air and between 400 and 600°C in argon. However, hardness and wear measurements indicate that DLN and Ti-DLN coatings are thermally stable up to 400°C in air with a high hardness and good wear resistance. In contrast, DLC coatings are stable only up to 300°C [7] in air, based on compositional and Raman spectroscopy. In non-oxidizing environments, DLC films are reported to be stable up to 400°C, based on Raman spectroscopy [12], and between 440 and 490°C, based on wear measurements [20]. Si-DLC [19] is reported to be stable up to 345°C for an extended period of time (245 h) in air. Silicon and oxygen containing a-C:H films are reported by Muller et al. [10] to be stable up to 600°C in an argon atmosphere, based on Raman spectroscopy. Thus, DLN coatings appear to exhibit a superior thermal stability than that of undoped DLC coatings, whereas they are similar to Si-DLC and Si–O-DLC coatings.

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Diamond-like amorphous carbon

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Available online 19 March 2002.

Abstract

Diamond-like carbon (DLC) is a metastable form of amorphous carbon with significant sp^3 bonding. DLC is a semiconductor with a high mechanical hardness, chemical inertness, and optical transparency. This review will describe the deposition methods, deposition mechanisms, characterisation methods, electronic structure, gap states, defects, doping, luminescence, field emission, mechanical properties and some applications of DLCs. The films have widespread applications as protective coatings in areas, such as magnetic storage disks, optical windows and micro-electromechanical devices (MEMs).

Author Keywords: Diamond-like carbon; Properties; Applications

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